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IS 1060-2 (1960): Methods of sampling and test for paper and allied products: Part 2 [CHD 15: Paper and its products]



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“Knowledge is such a treasure which cannot be stolen”

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IS : 1060 (Part II) - 1960

Indian Standard

METHODS OF SAMPLING AND TEST FOR
PAPER AND ALLIED PRODUCTS, PART II

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BUREAU OF INDIAN STANDARDS

MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

*Indian Standard***METHODS OF SAMPLING AND TEST FOR
PAPER AND ALLIED PRODUCTS, PART II****Paper Sectional Committee, CDC 15***Chairman***SHRI C. A. SUBRAHMANYAM**

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IS : 1060 (Part II) - 1960

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Indian Standard

METHODS OF SAMPLING AND TEST FOR PAPER AND ALLIED PRODUCTS, PART II

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 7 June 1960, after the draft finalized by the Paper Sectional Committee had been approved by the Chemical Division Council.

0.2 A number of test methods for paper and allied products has been prescribed in* IS : 1060 (Part I)-1956 Methods of Sampling and Test for Paper and Allied Products, Part I. They cover the more general types of tests required for paper and allied products. Tests included in this standard are for special purposes.

0.3 This standard has been based partly on the standards now in use in Defence Laboratories. Note has also been taken of the data available with the Government of India Stationery Office, and the Cellulose and Paper Branch of the Forest Research Institute. Due weightage had to be given to the need for international co-ordination among standards prevailing in different countries of the world, and these considerations led the Sectional Committee responsible for the preparation of this standard also to draw upon the methods of tests prescribed by the British Standards Institution, the American Society for Testing Materials and the Technical Association of the Pulp and Paper Industry, USA.

0.4 This general standard of test methods is a necessary adjunct to standard specifications for individual types of paper and allied products.

0.4.1 Test methods not included in this standard and not otherwise provided in the individual detailed standards shall be subject to agreement between the purchaser and the vendor.

0.5 This standard requires reference to the following Indian Standards:

*IS : 250-1953 SPECIFICATION FOR POTASSIUM BICHROMATE, TECHNICAL AND ANALYTICAL REAGENT

*IS : 266-1950 SPECIFICATION FOR SULPHURIC ACID

IS : 376-1952 SPECIFICATION FOR SODIUM HYDROXIDE, ANALYTICAL REAGENT

*IS : 460-1953 SPECIFICATION FOR TEST SIEVES (WITH AMENDMENT No. 1, NOVEMBER 1955)

*Since revised.

***IS : 1060 (PART I)-1956 METHODS OF SAMPLING AND TEST FOR PAPER AND ALLIED PRODUCTS, PART I**

IS : 1070-1960 SPECIFICATION FOR WATER, DISTILLED QUALITY (*Revised*)

0.5.1 Wherever a reference to any Indian Standard mentioned in **0.5** or otherwise, except IS : 460-1953,* appears in this standard, it shall be taken as a reference to the latest version of the standard.

0.6 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960 Rules for Rounding Off Numerical Values (*Revised*). The number of significant places retained in the rounded off value should be the same as that of the specified value in the standard, it being understood that the analyst will carry out his determination to at least one place more than in the specified value.

1. SCOPE

1.1 This standard prescribes methods of tests for paper for special purposes not covered in IS : 1060 (Part I)-1956.* The tests prescribed are:

- a) Bursting strength, wet;
- b) Tensile strength, wet;
- c) Bending test;
- d) Cupping test;
- e) Exudation test;
- f) Bleeding resistance;
- g) Fastness to light;
- h) Brightness;
- j) Water vapour permeability;
- k) Estimation of alpha cellulose content;
- m) Estimation of lead and lead compounds;
- n) Estimation of chlorides (water soluble);
- p) Estimation of sulphates (water soluble);
- q) Estimation of fatty and/or rosin acid extractable by ether;
- r) Estimation of matter soluble in ether; and
- s) Estimation of benzene soluble matter.

1.2 Should any inconsistency exist between the requirements of this standard and those of the standard for an individual material, the latter shall prevail.

*Since revised.

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions given in IS : 1060 (Part I)-1956* shall apply.

3. SAMPLING

3.1 Representative samples for test shall be drawn as prescribed in 3 of IS : 1060 (Part I)-1956*.

4. CONDITIONING

4.1 Conditioning of test specimens shall be carried out in accordance with 5 of IS : 1060 (Part I)-1956*.

5. QUALITY OF REAGENTS

5.1 Unless otherwise specified, pure chemicals and distilled water (see IS : 1070-1960) freshly boiled and cooled, shall be employed in tests.

NOTE— ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

6. BURSTING STRENGTH, WET

6.1 General — The popularity of bursting strength test depends not only on the ease with which the test is made, but also on the combination of strength, 'give' and toughness which it measures and which serve as a measure of the serviceability of paper in various applications. It has the disadvantage, however, that it depends in a complicated way on the machine direction tensile strength, stretch and the size of the burst area. Also it does not give any indication of cross direction tensile strength.

6.2 Equipment — A tester in which testing is done by hydraulic pressure communicated through the medium of glycerine or compressed air to a pure gum rubber diaphragm of thickness 0.85 ± 0.05 mm in contact with the paper shall be used. The gauge used shall be so chosen that the individual reading shall not be less than 25 percent or more than 75 percent of the total indicated capacity of the gauge. The gauge should be of the maximum reading type and should be calibrated not less frequently than once every six months.

6.3 Procedure — Immerse the test piece in water at $27^{\circ} \pm 2^{\circ}\text{C}$ for four hours. Remove and place it flat and straight on a pad of four thicknesses of dry blotting paper and cover it with a similar pad. Roll a metal cylinder weighing 500 ± 5 g and having a diameter of about 50 mm steadily over the top blotter to remove the surface moisture as quickly as possible. Immediately clamp the test specimen firmly (without slippage during the test) over the diaphragm, between two annular plane unpolished (matte) surfaces

*Since revised.

of 30.0 ± 0.5 mm internal diameter. Run the machine so that the pressure increases at a uniform rate of approximately 0.75 kg/cm^2 per second until the test piece bursts. Note from the pressure gauge the pressure in kg/cm^2 at which the test piece bursts. Take one reading with the wire side uppermost and one with the top side uppermost with each sample sheet.

NOTE — A rate of 120 revolutions per minute in glycerine-operated machine is usually satisfactory.

6.4 Report — Report the type of tester used and give the average, maximum and minimum values of the reading for each side separately.

6.5 Burst Factor — Used for comparing two papers with regard to their bursting strength:

$$\text{Burst Factor} = \frac{\text{Bursting strength in g/cm}^2}{\text{Substance in g/m}^2} \text{ [see 6 of IS : 1060 (Part I) - 1956*]}$$

7. TENSILE STRENGTH, WET

7.1 General — This method of test covers a procedure for determining the tensile strength of paper and paper products after wetting with water or other liquid. It is of special usefulness in evaluating the performance characteristics of paper and paper products subjected to stress during processing or use, while wet. Depending on the requirement in use, the test piece may be immersed in the appropriate liquid for a given time and tested whether it is completely saturated or not. However, unless otherwise specified, the wet tensile strength test is assumed to be performed on the test piece after complete saturation in water.

7.2 Test Piece — Prepare a sufficient number of test pieces cut from each principal direction of the paper, excluding watermarks, creases or other visible imperfections. The pieces shall be clean-cut to within plus or minus one percent of their nominal widths, with edges parallel, and shall be long enough to be inserted in the jaws of the clamps of the testing apparatus without handling the section under test. The width of the test pieces shall not exceed 37.5 mm nor shall be less than 12.5 mm.

NOTE — Varying the width of the test pieces between 12.5 and 37.5 mm, with a proportionate change in the rate of loading, does not, in general, make much difference in the proportionate test results except for unbeaten long-fibre papers, when the difference may be appreciable.

7.3 Procedure

7.3.1 Use the testing machine referred to in 12.4 of IS : 1060 (Part I) - 1956* and calibrate as follows:

Accurately level the apparatus in both its principal directions and make the mechanism move freely. Apply weights of known value to the upper clamp so that it moves in the same direction and plane

*Since revised.

as when testing. Disengage the pawls of the pendulum, suspend the test weight by a flexible connection from the centre of the upper clamp, release the latch holding the pendulum at zero indication, and allow the pendulum to come slowly to equilibrium. Note the indicated scale reading for each load applied and record all test data. It is recommended that readings be taken at not less than three distributed points on the scale of the instrument.

7.3.2 Immerse the test piece in the specified liquid till saturated. Normally 4 hours' immersion in water will be adequate for most papers. Unless otherwise required, the liquid shall be maintained at $27^{\circ} \pm 2^{\circ}\text{C}$.

7.3.3 At the end of the immersion time and before placing the test piece in the tensile testing machine, place it flat and straight on a pad of four thicknesses of dry blotting paper and cover it with a similar pad. Roll a metal cylinder weighing 500 ± 5 g and having a diameter of about 50 mm steadily over the top blotter, taking about 2 seconds for the operation.

7.3.4 Immediately after blotting, place the test piece loosely in the jaws of the clamps, align it squarely, then tighten the upper clamp, take up the slack, and finally tighten the lower clamp, without touching the portion under test. Then apply the load as follows:

For weak papers breaking below 2.5 kg per 15 mm width, adjust the loading so that fracture occurs in not less than 5 nor more than 15 seconds. For all other papers, adjust the rate of loading so that the lower clamp moves at a rate of 30.0 ± 1.5 cm/min.

7.3.5 At least ten test pieces from each direction of the sheet shall be tested.

7.4 Report — Report the average, maximum and minimum values of the test results. Express the tensile strength, wet, in kg/cm width to three significant figures and also as a percentage of the dry tensile strength. Express the stretch in each direction separately as percentage elongation correct to one place of decimal. The exact width of the test piece and the initial distance between the jaws shall be reported with the results.

8. BENDING TEST

8.1 General — This test is applicable to paperboards.

8.2 Procedure — Fold a square test piece at least 60×60 mm by fingers along the diagonal so that the inner surfaces are in contact completely. Unfold and repeat the folding along the same crease in the opposite direction until the other surfaces are in contact completely. Repeat the test with a second test piece.

8.2.1 Record any visible fibrous breaks or cracks on the surface of the folded test piece after each crease is made. Report the average number and length of the visible breaks on the surface and, if coated, the appearance of

the folded surface resulting from the test. If no break is more than 6 mm long, the board may be classified as fair. If the board shows no fibrous break, it is classified as good.

9. CUPPING TEST

9.1 General — This test is applicable to paperboards.

9.2 Procedure — Cut out a disc from each test piece. Damp the disc by dipping momentarily in water and then air dry for 5 to 10 minutes till all surface moisture is removed but the paper remains just damp. Then press the disc into a cup-shaped mould of diameter approximately 9 mm to form a cup 3 mm deep. The radius of curvature of the plunger shall be 0.5 mm.

9.2.1 The cup so produced must be free from chipping and cracks at the bottom where the material is turned up.

10. EXUDATION TEST

10.1 General — This method of test is applicable in the case of bitumen or pitch coal tar impregnated papers.

10.2 Procedure — Place the test piece of suitable size over and in contact with a clean white filter paper and apply a load of 6.4 kg through a flat metal plate of 45 cm² area. Maintain a temperature of $72^{\circ} \pm 2^{\circ}\text{C}$.

10.2.1 There should be no exudation of the impregnating material or staining of the filter paper at the end of two hours.

11. BLEEDING RESISTANCE TEST

11.1 General — This method covers the test for determining the degree of bleeding resistance which is a measure of the ability of bitumen or asphalt laminated or similarly treated case-liners or baling paper to avoid staining the contents of a case or bundle.

11.2 Apparatus — The apparatus shall consist of the following:

- a) *Pressure Block* — A block of metal 75 × 75 mm weighing about 2 kg with a smooth, flat under-surface. A wooden knob fastened to the top of the block conveniently serves as a handle.
- b) *Bed Plate* — A piece of metal 75 × 75 mm and about 6 mm thick, having a smooth, flat upper surface.
- c) *Plate Guide* — A guide for the pressure block which may conveniently be made from 115 × 115 mm sheet metal with the corners turned up vertically.
- d) *Metal Separators* — 75 × 75 mm made from thin, flat aluminium sheet or heavy aluminium foil.

- e) *Paper Sheets* — 75 × 75 mm cut from smooth, white imitation art paper.
- f) *Oven* — capable of maintaining the test assembly at a constant temperature of $66^{\circ} \pm 2^{\circ}\text{C}$.

11.3 Test Piece — The test piece shall be 75 × 75 mm cut in such a way as to be representative of the sample to be tested.

11.4 Procedure

11.4.1 Put the test pieces and the disassembled apparatus in the oven at 66°C for a sufficient time for all to reach that temperature. Place a test piece between the two sheets of white imitation art paper and place the combination between two aluminium separator sheets. If desired, up to five of such sandwiches may be laid on one another but separated by single metal sheets and tested simultaneously.

11.4.2 Place the sandwiches on the bed plate in the guide and cover with the pressure block (which exerts a pressure of 0.035 kg/cm^2). Make this assembly with as little cooling as possible. Leave the assembly in the oven at $66^{\circ} \pm 2^{\circ}\text{C}$ for 5 hours.

11.4.3 Then remove, separate the sandwiches and examine the white imitation art paper for staining by the exuded material.

11.5 Report — Report the degree of bleeding as follows, disregarding any staining arising from the cut edges of the test piece:

<i>Degree of Bleeding</i>	<i>Description</i>
a) No bleeding	No visible staining
b) Slight bleeding	Staining in the form of specks only; blotches or spots under 1.5 mm in diameter
c) Considerable bleeding	Staining in the form of spots and blotches 1.5 mm or more in diameter

NOTE — Care should be taken that radiant heat does not raise the temperature of the assembly considerably above the indicated temperature of the oven.

12. FASTNESS TO LIGHT

12.1 The fastness to light of the colour of the paper is tested in a Kelvin fugitometer.

12.2 Kelvin Fugitometer — This comprises a revolving drum which carries the samples under test in special holders. These sample holders are suspended from the top of the revolving drum in a constant upward stream

of air, conditioned to a predetermined humidity and temperature. This is assured by a horizontal fan which rotates just above the level of the humidifying water in a circular tray at table level. The temperature of this water is controlled by immersion heaters with thermostatic controls.

12.2.1 The source of fading radiation is the standard carbon arc lamp*. On comparison of spectrum of the fugitometer lamp with that of sunlight, there is very close resemblance — particularly at the violet end of the spectrum — and because of the steady and intensive light impinging on the samples under test, the period of time required to produce results comparable with natural conditions of sunlight is substantially reduced.

12.3 Procedure — Expose the test piece in the fugitometer for the period specified in the specification for the individual material. The test piece passes the test if no fading occurs.

13. BRIGHTNESS

13.1 General — This method is intended for determining the $45^\circ - 0^\circ$ directional reflectance of uncoloured paper for blue light. In the paper trade, this quantity is known as 'brightness'.

NOTE — Brightness, as determined by this test is primarily a measure of freedom from yellowness arising in pulp and paper from the presence of lignin and other so-called impurities left by incomplete bleaching. It thus differs from luminous (visible) reflectance. Blue light is used for the measurement of brightness, whereas light of all colours, with major weight given to the green is used to measure luminous reflectance.

13.2 Apparatus — The apparatus shall consist of a reflectometer, either visual or photo-electric type, having such source, filter, and receptor characteristics that it will measure, for undyed papers and pulps, $45^\circ - 0^\circ$ directional reflectance for blue light as herein described, accurate within 1.0 percent. The apparatus shall have the following characteristics:

- a) *Spectral Characteristics* — The product of spectral energy of source, spectral transmission of any filters through which light must pass, and spectral response of receptor shall be equivalent to the product of the z function of the standard observer and colorimetric co-ordinate system recommended by the International Commission on Illumination† multiplied by the energy distribution of the Commission's standard illuminant A (representative of incandescent lamp light). Such a combination will have a maximum spectral response to energy at about 460 millimicrons.

*Electrical particulars of a typical fugitometer are:

Power supply	...	50 cycles ac	Arc voltage	...	120-145 V
Line voltage	...	200-250 V	Arc current	...	15-17 A

†Proceedings of eighth session of the International Commission on Illumination, Cambridge, England, September 1931.

- b) *Geometric Characteristics* — Rays incident on the test piece shall spread no more than 4° from the axis of the incident beam; rays accepted for measurement shall spread no more than 15° from the axis of the viewed beam. Rays in the incident and viewing beams shall be symmetrically arranged with respect to their respective axes. The above directions of illuminating and viewing may be inter-changed, giving the equivalent $0^\circ - 45^\circ$ conditions.

13.3 Standards

13.3.1 Primary Standard — The primary standard of $45^\circ - 0^\circ$ directional reflectance shall be a layer (at least 0.5 mm in thickness) of magnesium oxide freshly prepared by collecting the smoke from burning magnesium ribbon or turnings on a suitable surface in accordance with Appendix A.

13.3.2 Working Standards — Either of two types of working standards may be used for day-to-day operation of the instrument : (a) pieces of paper carefully measured on an instrument known to be in good adjustment, and (b) ceramic tiles or porcelain-enamelled plaques carefully measured on an instrument known to be in good adjustment and to possess essentially the same geometry as the device with which they are to be used. Calibrated pieces of paper shall be used for only a short period following their calibration, and soiled pieces shall be discarded. Ceramic tiles and porcelain enamelled plaques, usually can be cleaned without alteration of their reflective properties. Nevertheless, working standards of this second type should be checked from time to time for assigned values of reflectance for blue light. The assigned reflectance for blue light of the working standard shall be calculated as follows:

$$R_1 = \frac{b}{a} \times 100$$

where

R_1 = assigned reflectance of the working standard, in percent;

b = average reading for working standard; and

a = average reading for primary standard.

13.4 Test Piece — Test pieces shall be handled carefully to avoid soiling, and care shall be taken not to touch the areas tested. A pad of test pieces shall be used for the measurement, so thick that doubling the number of sheets does not cause change in the measured reflectance.

13.5 Procedure

13.5.1 When available, a working standard of about the same reflectance for blue light as the test pieces shall be used. The reflectance of this working standard shall be read both before and after readings of reflectance of the

test pieces are made. Readings shall be made on at least five separate test pieces in each pad. To average out any effects of rotation, equal number of readings shall be taken along and across the machine direction.

13.5.2 The $45^\circ - 0^\circ$ directional reflectance shall be calculated as follows:

$$R = \frac{c}{b} \times R_1$$

where

$R = 45^\circ - 0^\circ$ directional reflectance for blue light of the sample, in percent;

c = average reading for test pieces;

b = average reading for working standard; and

R_1 = assigned reflectance of working standard; in percent.

13.6 Report — Values of $45^\circ - 0^\circ$ directional reflectance for blue light shall be reported for the wire and felt sides separately, to the nearest one percent. The report shall state that this method was used.

NOTE — Because of the variability of standard magnesium oxide surfaces, possible inaccuracies of photometers, possible inaccurate spectral conditions, geometric conditions, etc, it is felt that results accurate to better than about plus or minus one percent are not obtainable. However, where parties interested in a test are willing to accept values assigned to a working standard as accurate to plus or minus 0.1 percent, results can be obtained that can be reproduced to plus or minus 0.2 percent, or even 0.1 percent.

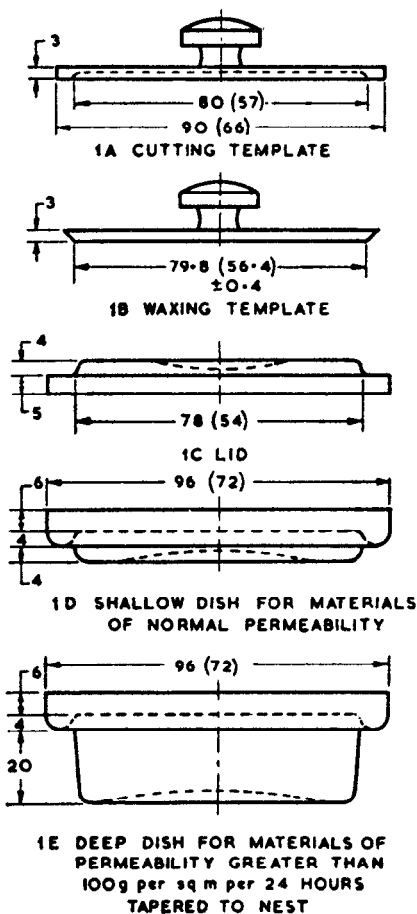
14. WATER VAPOUR PERMEABILITY TEST

14.1 General — This method is for the determination of water vapour permeability of paper and board under 3 mm thickness, under prescribed conditions of temperature and humidity. A quantity of desiccant, enclosed in a dish sealed by a sheet of the material, is stored in a conditioned atmosphere and the rate of water vapour transmission is computed from the rate of increase in weight of the dish assembly.

14.2 Apparatus — The apparatus shall consist of the following:

- a) *Test Dishes* — Shallow aluminium dishes of as large a diameter as can conveniently be accommodated on the balance, should be used. The test dishes shall be designed so that the area of a test piece is accurately defined and a wax seal between the test piece and the dish satisfactorily prevents the transmission of water vapour at or through the edges of the sheet. Figure 1 shows a suitable design of dish and templates for cutting and sealing the test pieces. Using the dimensions shown, test areas of 50 cm² and 25 cm² are obtained. The dishes and their covers may be manufactured from 0.900 mm deep drawing quality aluminium sheet.

- b) *Weighing Covers* — If the test dishes are removed from the cabinet or room for weighing, an aluminium lid (see Fig. 1) should be provided to fit each dish in order to minimize changes in weight while the test pieces are out of the controlled atmosphere.



All dimensions in millimetres.

NOTES — 1. Dimensions are shown for test areas of 50 cm² and 25 cm², the latter in brackets.

2. Figures for dishes and lids show inside dimensions, except the overall diameter of the dishes, which is an outside dimension.

FIG. 1 DIAGRAM OF DISHES AND TEMPLATES

- c) *Balance* — The balance used shall be accurate to 0.000 5 g.
- d) *Humidity Cabinet* — The cabinet shall have racks to support the dishes and means of circulating air-conditioned at:
 65 ± 2 percent relative humidity and $27^\circ \pm 2^\circ\text{C}$, or
 90 ± 2 percent relative humidity and $38^\circ \pm 1^\circ\text{C}$
continuously over the test pieces. The air circulation shall be sufficiently rapid to maintain the specified humidity at the surface of test piece. The design of the cabinet shall be such that no condensation of moisture takes place in the vicinity of the test pieces during the test. Care shall be taken to ensure that the cabinet is not loaded to such an extent that the rate of moisture up-take reduces the relative humidity below the lower limit specified.
- e) *Desiccant* — The desiccant used shall be such that the relative humidity within the dish does not rise above 2 percent during the determination. Anhydrous calcium chloride and magnesium perchlorate are suitable and shall be used in the form of small lumps passing through IS Sieve 240 ($2\,399 \pm 91$ microns, see IS : 460-1953*) but free from fines passing through IS Sieve 60 (592 ± 33 microns, see IS : 460-1953*).
- f) *Wax* — The wax compound used for sealing shall adhere firmly both to the surfaces of the test piece and to the dish and shall not be brittle at room temperatures. It is important that the wax be relatively stable and non-absorbent to water vapour ; the weight change on an area of 50 cm² of wax surface exposed for 24 hours at 90 percent relative humidity and $38^\circ \pm 1^\circ\text{C}$ shall be less than 0.001 g.

14.3 Test Piece — The sides of the test piece shall be designated I and II respectively, side I being that side which in use is exposed to the higher humidity whether this be inside or outside the package. Where the conditions of use of the material are unknown, side I shall be that side which faces outwards. If the material is coated on one side only, the word coated shall be added to the designation, for example, Side I (coated).

14.3.1 The determination shall be made with test pieces as follows:

- a) Uncreased with side I exposed to the high humidity,
- b) Uncreased with side II exposed to the high humidity,
- c) Creased with side I exposed to the high humidity, and
- d) Creased with side II exposed to the high humidity.

A total area of not less than 100 cm² made up of at least two test pieces shall be examined for each of the four types of tests listed above. The minimum area of a test piece shall be 25 cm².

*Since revised.

14.4 Creasing

14.4.1 Creasing shall be done in the conditioning atmosphere ($27^{\circ} \pm 2^{\circ}\text{C}$, and 65 ± 2 percent RH) or immediately after removal therefrom.

14.4.2 Cut square samples and, after conditioning, crease to give two sets of pleats, equal in number, along the two principal directions of the sheet. Use a flat plate as the creasing bed and make the creases by a loaded platen whose width is about 2 cm greater than the length of the sample to be creased. The creasing load shall be one kilogram per centimetre of crease and shall be applied for 10 to 15 seconds. The total length of crease in centimetres shall be equal to the effective area of the test piece in square centimetres.

14.4.3 Creases shall be made one at a time and immediately unfolded, adjacent creases being made on opposite sides of the sheet so that the final effect is a set of pleats. The process shall be repeated to produce a similar set of pleats at right angles to the first. A circular test piece shall then be cut from the square creased sample.

14.4.4 The spacing between adjacent creases shall be as below:

1.85 cm for an effective test area of 25 cm^2 , and

2.10 cm for an effective test area of 50 cm^2 .

14.5 Procedure

14.5.1 Cut the test piece using a template which is of such diameter that the edge of the test piece covers half the annular recess of the dish. Fill the dish with desiccant to within 1 to 2 mm of the supporting ring. Place the test piece on the supporting ring and centre (*see* Fig. 2). Place the waxing template centrally over the dish and test piece, and run molten wax into the annular recess until the wax is level with the top surface of the template. Break any air bubbles in the wax with a small gas jet, allow the wax to harden and remove the template. Inspect the dish to ensure that the seal is satisfactory and remove any wax on the outside. It is important that the filling and sealing of the dish is carried out as rapidly as possible so that the desiccant absorbs a minimum of water vapour from the atmosphere. Care must be taken not to damage the test area during the operation or to allow the desiccant to come into contact with it. To facilitate the removal of the template from the wax, apply a thin film of petroleum jelly to the bevelled edge before sealing; remove any surplus on the lower surface.

14.5.2 Prepare the required number of dishes and place in the humidity cabinet. Make successive weighings at suitable intervals which must be sufficiently frequent to complete the test before the relative humidity in the dish rises above 2 percent. Plot the cumulative weight increase of each dish in milligrams against the total time of exposure in the cabinet. When at least three, but preferably four, points lie on a straight line, a constant

rate of gain has been attained and the experiment may be discontinued. The weighings should be made to an accuracy of one percent of the weight change between two successive weighings or 0.001 g whichever is larger.



FIG. 2 CENTRING SAMPLE ON DISH

14.5.3 If the samples are exposed in a cabinet from which they are removed for weighing, precautions must be taken to ensure that the dishes are out of the cabinet for the same period and under the same conditions at each successive weighing. The following procedure is recommended. About eight dishes are removed from the cabinet at a time and immediately covered with their respective lids. The dishes should be allowed to cool until their temperature is not appreciably different from that of the room. At the completion of the weighings, the lids are removed and the samples immediately replaced. Precisely the same routine and time schedule must be followed in detail at each successive weighing interval. Where possible, the cabinet and balance should be kept in a room having constant humidity and temperature.

14.6 Report

14.6.1 Report the water vapour permeability as g/m^2 per 24 hours at the temperature and relative humidity specified for the material.

14.6.2 Calculate the permeability from the slope of the graph which has been obtained by plotting the cumulative weight increase of each dish in milligrams against the total time of exposure in the cabinet. The required slope is that of the best straight line drawn through those points which represent the constant rate of gain. If it is such that x mg is the weight increase over a period of y hours for an exposed area of A cm², the permeability is $\frac{240x}{Ay}$ g/m² per 24 hours.

14.6.3 The average, maximum and minimum values for each type of test made as in 14.3.1 shall be reported to two significant figures or the nearest 0.2 g/m² per 24 hours, whichever is larger.

15. ALPHA-CELLULOSE CONTENT

15.1 General — Alpha-cellulose is taken as the fraction that can be filtered out of a mixture consisting of the fibrous material and sodium hydroxide solution of maximum dissolving power (7.3 percent, w/w), after the fibres have previously been swelled with sodium hydroxide solution (17.5 percent, w/w or 5.24 ± 0.05 N). After separation, the alpha-cellulose is determined either by drying and weighing, or volumetrically by oxidation with potassium dichromate. Both methods are capable of the same reproducibility and give practically the same values. In the volumetric method, no moisture or ash determinations are made and a much smaller sample is used, resulting in a shorter and more rapid procedure.

15.2 Apparatus — consisting of the following:

- a) *Grinder* — A Koerner or equivalent type grinder that will completely disintegrate the paper without heating or contaminating it.
- b) *Mixer* — A device with which uniform mixture of the ground material can be secured. A mixer is easily constructed by fitting a crock or bell jar, of approximately equal diameter and depth, with a wooden lid through which runs the shaft of an electric fan, vanes being properly placed to produce turbulence. Mixing for 20 to 40 seconds at full speed normally suffices; longer periods result in separation of the light material from the heavy.
- c) *Bath* — A water-bath that can be maintained at $20.0^\circ \pm 0.1^\circ\text{C}$.
- d) *Electrometric Titration Apparatus* — For the estimation of dichromate, an indicator may be used as described in 15.3(e), but for rapid, accurate analysis, an electrometric apparatus is recommended. Any ordinary potentiometric circuit with a platinum wire electrode and calomel half-cell is suitable. A suitable arrangement is shown in Fig. 3. The potentiometric arrangement may consist of a galvanometer with a sensitivity of 0.5 to 1.0 microampere per millimetre scale division, a dry cell, and a sliding-contact rheostat having a

total resistance of approximately 400 ohms. Smaller rheostats will suffice but they drain the dry cell more quickly. The large deflection at the end point is unmistakable from possible slow creeping during the titration. The galvanometer is adjusted to zero by varying the resistance at the beginning of the titration.

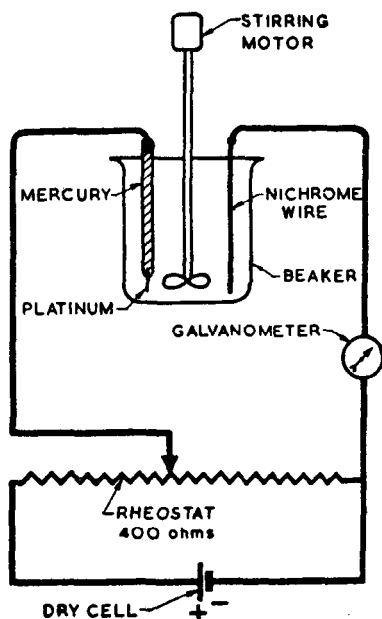


FIG. 3 ARRANGEMENT FOR ELECTROMETRIC TITRATION

15.3 Reagents — consisting of the following:

- Sodium Hydroxide Solution** (17.5 percent, w/w or 5.24 N) — Allow a 50 percent solution of sodium hydroxide (conforming to IS: 376-1952) to stand about one week in a stoppered vessel to permit settling of sodium carbonate. Draw off 2.0 ml of the supernatant liquid with a pipette, and about 50 ml of distilled water and 1 ml of 3 N barium chloride to lessen the effect of carbon dioxide on the end point, and titrate with standard 1 N hydrochloric acid, using phenolphthalein as indicator. Knowing the approximate normality of the concentrated sodium hydroxide, dilute it with distilled water to 5.24 ± 0.05 N, checking the diluted sodium hydroxide by titrating 10.0 ml of it as before, and diluting further, if necessary, to obtain the normality specified.
- Potassium Dichromate Solution** — Dissolve 90.0 g of oven-dry (100° to 105°C) potassium dichromate (conforming to IS: 250-1953*) in

*Since revised.

hot water (70° to 90°C), and dilute to one litre after allowing the solution to cool.

- c) *Ferrous Ammonium Sulphate Solution* — Dissolve 195 g of ferrous ammonium sulphate crystals in water containing 10 ml of sulphuric acid (sp gr 1·84) and dilute to one litre. If the solution is kept out of contact with oxygen, for example, by means of a slow, continuous stream of hydrogen, its strength will remain quite constant. The amount of hydrogen thus used is about one-fifth of a 5 m³ cylinder per year. This precaution is not necessary, but reduces the frequency with which the potassium dichromate-ferrous ammonium sulphate ratio must be determined from once a day to two or three times a month.
- d) *Acetic Acid Solution for Gravimetric Procedure* — Prepare a solution of acetic acid approximately 10 percent by weight.
- e) *Dichromate Indicator* — If electrometric apparatus is not available, dissolve 0·3 g of barium diphenylamine sulfonate and 0·5 g of sodium sulphate in 100 ml of water. Use 5 to 10 drops in a solution sufficiently dilute for good observation of the end point, which is shown by a change of colour from red to green. As an alternative, the outside indicator, potassium ferricyanide solution, may be placed in drops upon white porcelain. The end point occurs when a drop of the solution being titrated turns a drop of the ferricyanide indicator blue.
- f) *Barium Chloride (3 N)* — Dissolve 37 g of barium chloride crystals in water and dilute to 100 ml.
- g) *Sulphuric Acid (24 N)* — Cautiously add three volumes of sulphuric acid of sp gr 1·84 (conforming to IS : 266-1950^{*}) to two volumes of water in a heat-resistant flask, cool by tap water, and add the acid in small portions, shaking after each addition. The acid will then be approximately 72 percent by weight.
- h) *Sulphuric Acid (6 N)* — Dilute one volume of the 24 N sulphuric acid to 3·5 volumes in a graduated cylinder. After mixing, standardize against the 5·24 N sodium hydroxide and then dilute to $6·0 \pm 0·1$ N.

15.4 Test Piece — The test piece shall be cut from the specimen obtained as in 3 of IS : 1060 (Part I)-1956^{*} in such a way as to be representative of the lot. It shall be reduced to cotton-like form in the disintegrator and then thoroughly mixed. If the paper is mineral-coated, the coating shall be removed before grinding.

15.5 Test Temperature — Perform all operations (except weighing in 15.6.1) and keep all liquids at $20·0^\circ \pm 0·1^\circ\text{C}$.

^{*}Since revised.

15.6 Volumetric Method

15.6.1 Weigh 0.30 ± 0.01 g of the disintegrated test piece in a 100-ml beaker. Add 20.0 ml of sodium hydroxide (17.5 percent), macerate until the fibres are uniformly wet and dispersed, and let it stand for 10 minutes from the time of addition of sodium hydroxide. Then add 33 ml of water, stir the mixture thoroughly, and let it stand for one hour more, stirring once during the interval. After stirring once more, pour about 5 ml of the unsettled mixture on a copper or brass wire screen of 0.177 ± 0.011 mm aperture (IS Sieve 18, see IS: 460-1953) fitted into a Gooch crucible. The crucible and ring are supported by a funnel fitted into the neck of a 100-ml volumetric flask with a rubber stopper through which passes a glass tube for suction (pressure differential 10 to 20 mm of mercury). Avoid excessive packing of the fibres, as this retards filtering. It may be necessary to re-filter the first filtrate, but loss of small amounts of alpha-cellulose to the filtrate does not affect the results appreciably. Pour the remainder of the mixture on the mat and, before the last of the liquid has run through, wash the beaker and the mat with 35 ml of water.

15.6.2 Moisten the residue of alpha-cellulose with water and remove it from the crucible. Place the crucible upright in a 400-ml beaker, fill it with 25 ml of 24 N sulphuric acid at room temperature, and rinse it after a few minutes with 50 ml more of the acid. Disintegrate the alpha-cellulose pad in the acid, using a stirring rod. Add to the alpha-cellulose solution, with a pipette, 25.0 ml of the potassium dichromate solution, and heat at 140° to 150°C for 10 minutes. Bubble air in a fine stream through the solution to prevent bumping, and keep the beaker covered with a watch glass, notched to permit entrance of a thermometer and the bubbling tube. After the solution has cooled to 130°C , add 50 ml of water, rinse the thermometer, bubbling tube, etc, and cool the solution to 60°C or lower. Titrate the remaining potassium dichromate with the ferrous ammonium sulphate solution.

15.6.3 Pipette exactly half of the filtrate from the alpha-cellulose, after all fibres present have settled, into a 400-ml beaker containing 5.0 ml of potassium dichromate solution. If the paper contains oxidizable fillers, such as zinc sulphide pigment or calcium sulphite, filter the filtrate once through a thick pad of asbestos in a Gooch crucible before taking the portion for analysis. (Such fillers remaining with the alpha-cellulose may cause some error, but this is usually slight.) Cautiously, while stirring constantly, pour 50 ml of sulphuric acid (sp gr 1.84) down the side of the beaker containing the portion of the filtrate for analysis, then heat and titrate as described in 15.6.2 for the alpha-cellulose solution.

15.6.4 Calculation — Calculate the percentage of alpha-cellulose from the following equations, except that when rosin, starch, or glue are present, corrections shall be made in accordance with 15.6.4.1:

$$A = 25 - (V_1 \times r) \quad \dots \quad (1)$$

$$B = 2 \times (5 - V_2 \times r) \quad \dots \quad (2)$$

*Since revised.

$$\text{Alpha-cellulose, percent} = \frac{A}{A + B} \times 100 \quad \dots \quad (3)$$

where

A = volume of potassium dichromate solution in ml required to oxidize the alpha-cellulose;

B = volume of potassium dichromate solution in ml required to oxidize the filtrate;

V_1 and V_2 = volume of ferrous ammonium sulphate solution in ml required for titration of the potassium dichromate remaining after oxidation of the alpha-cellulose and filtrate, respectively; and

r = volume of potassium dichromate equivalent to one millilitre of ferrous ammonium sulphate solution (determined frequently by titrating 5 ml of potassium dichromate in 100 ml of diluted sulphuric acid (1 : 1)).

15.6.4.1 If rosin, starch, or glue are present, the volumes of potassium dichromate shall be corrected as follows before calculating the percentage of alpha-cellulose:

The amounts of sizing materials remaining with the alpha-cellulose are taken as 0.25 percent glue, 0.2 percent starch, and 0.2 percent rosin, based on the dry weight of the sample (see Note). Convert these values into weights, and then into millilitres of the potassium dichromate solution by dividing each weight by the corresponding factors, which are 0.015 4 g/ml for glue, 0.012 9 g/ml for starch, and 0.006 6 g/ml for rosin. Subtract the resulting volumes in millilitres of the potassium dichromate solution from the volumes of potassium dichromate consumed by the alpha fraction and substitute the corrected values in equations 1 and 2.

NOTE—These are average values but actual amounts have been found to vary not more than 0.1 percent from them, irrespective of the content of glue, starch, or rosin in the papers. For example, if a paper were found to contain 3.4 percent glue, 0.7 percent starch, and 1.1 percent rosin, then the sizing materials remaining in the beta plus gamma portion would be 3.15, 0.5, and 0.9 percent respectively.

15.7 Gravimetric Method

15.7.1 Allow the disintegrated test piece to come to moisture equilibrium with the atmosphere of the balance case. Weigh, to the nearest milligram, 1.5 g of the disintegrated test piece for the alpha-cellulose determination. Weigh at the same time samples for the moisture and ash determinations, and also for those determinations of components other than cellulose as may be found necessary for calculation of the total cellulose content, such as fillers and sizing materials.

15.7.2 Add 100 ml of sodium hydroxide (17.5 percent, *w/w*) to the weighed quantity of the test piece in a 400-ml beaker. Macerate until uniformly wet and dispersed, and let it stand for 10 minutes from the time of addition of sodium hydroxide. Dilute with 165 ml of water, stir the mixture thoroughly, and let it stand for one hour more, stirring once during the interval. After stirring once more, pour the mixture upon a medium-weight cotton cloth cut to fit a 75 mm Buckner funnel, the cloth having been previously washed, and the fitted piece dried to constant weight at 100° to 105°C and weighed in a weighing bottle before use. Remove by refiltration any fibres passing through the filter. Before the last of the liquid has run through, add water, break up the pad well with a pointed stirring rod, and keep it in a loose condition until 200 ml of wash water have passed through. Then cover the alpha-cellulose with acetic acid (10 percent) and allow to soak for 5 minutes after which pass an additional 500 ml of wash water through while breaking up the pad with a pointed stirring rod.

15.7.3 Dry the alpha-cellulose on the cloth in the original weighing bottle overnight at 100° to 105°C, cool, and weigh. Determine the ash content and correct the weight accordingly. The sizing materials, if any, remaining with the alpha-cellulose are taken as 0.25 percent glue, 0.2 percent starch, and 0.2 percent rosin, based upon the dry weight of the test piece, and the corresponding weights subtracted from the total.

15.8 Report — The percentage of alpha-cellulose shall be based upon the total cellulose including pentosans, but excluding moisture, ash, rosin, or any sizing or other added nonfibrous materials. All determinations of alpha-cellulose shall be made in duplicate, the results of which shall agree at least within 0.4 percent of the dry weight of the sample, and the average shall be expressed to the nearest one percent. The report shall state whether the volumetric or the gravimetric method was used.

NOTE — The precision of the volumetric method is much greater than these requirements would indicate. It is felt, however, that the average value of two determinations which differ by 0.4 when expressed as a percentage, adequately represents the specimen as prepared for testing. When, however, a given specimen is ground and tested in different laboratories, greater disagreement may arise, due to the differences in freeness and extent of fibrillation of the fibres on the one hand, and individual differences in analysis on the other, added to the lack of perfect uniformity of the test specimen, and ordinary errors in analysis. Therefore, the reporting of alpha-cellulose values to the nearest one percent is more compatible with experience, and is sufficient for the practical usefulness of the value.

16. ESTIMATION OF LEAD AND LEAD COMPOUNDS

16.1 General — The method of test for the estimation of lead covers all papers and paper products where lead restriction is specified.

16.2 Procedure

16.2.1 Weigh 20 g of the material into a silica basin and carefully ash. On no account shall the basin get above a faint red heat, otherwise lead may be lost by volatilization. Extract the ashed material for one hour with 1 : 3 nitric acid and filter. Now add 2 ml of concentrated sulphuric acid to the nitric acid solution in a 150-ml beaker and evaporate to fuming on a hot plate. Add 100 ml of water and keep the beaker on the water-bath for 15 minutes, then at the room temperature overnight, and filter.

16.2.1.1 The lead sulphate must be separated from other insoluble material, such as barium sulphate, silica, etc, as in **16.2.2**.

16.2.2 Transfer the filter paper and the residue to a small beaker and cover with about 20 ml of water. Add 1 to 2 g of solid ammonium acetate, allow to stand on the water-bath for at least half an hour stirring occasionally. Then decant the liquid through a No. 42 Whatman filter paper or equivalent and repeat the extraction. Wash the beaker into the filter with warm water a few times to make sure that all the lead solution is extracted. Collect the filtrate in a 150-ml beaker. Pass hydrogen sulphide for 10 to 15 minutes and filter the lead sulphide at once and wash thoroughly but quickly with hydrogen sulphide water, the filtrate and washings being retained. Transfer the precipitate and paper to a tared crucible, carefully ignite, sulphate the ash, cool and weigh.

16.3 Report — Lead is calculated as below :

$$\text{Lead (as Pb), percent by weight} = \frac{A \times 0.683}{B} \times 100$$

where

A = weight of lead sulphate residue obtained ; and

B = weight of the material taken for estimation.

Two determinations shall be made and reported.

17. ESTIMATION OF CHLORIDES (WATER SOLUBLE)

17.1 General — This method is applicable to papers containing small quantities of chlorides soluble in water. Since the amounts to be determined are small, it is important that the determinations should be carried in a chloride free atmosphere.

17.2 Reagents — The following reagents are required:

- a) *Standard Silver Nitrate Solution* — Dissolve 2.5 g of silver nitrate and dilute to one litre. Standardize on 0.03 g portions of sodium chloride, by dissolving it in water and titrating as described below:

Adjust volume so that one millilitre of silver nitrate solution is equivalent to 0.000 838 g of sodium chloride.

b) *Potassium Chromate Indicator* — approximately 5 percent.

17.3 Procedure

17.3.1 Weigh out 5 g of the test piece and transfer to a 500-ml Erlenmeyer flask. Add 250 ml of boiling distilled water, attach a water or air condenser to the flask and boil gently for one hour. A ground glass connection between the condenser and flask should be used to avoid contamination. Filter the contents of the flask through a Buckner funnel. Return the paper sample to the flask. Add another 200 ml of boiling water to the paper and again extract for 15 to 30 minutes. Filter through the same filter previously used and wash the extracted specimen and filter with 50 ml of hot water in small portions. Run blanks on the water for each series of tests at the same time, putting the blanks through the same operations as the test pieces.

17.3.2 Combine the filtrates. If the extract is not neutral to litmus, adjust to neutrality with very dilute nitric acid or chloride-free sodium hydroxide. Add 5 ml of 5 percent potassium chromate solution as indicator and titrate with the standard silver nitrate solution to the first, brick red colour of silver chromate. Carry out a blank determination of the same amount of water as used in extraction of the paper and subtract the volume of silver nitrate required for the blank from that used for the titration of the paper extract. From the difference, calculate the percentage of chlorides, as sodium chloride, on the conditioned paper.

17.4 Result — Calculate as below:

$$\text{Chlorides (as NaCl), percent by weight} = \frac{A \times 0.000838}{B} \times 100$$

where

A = volume of silver nitrate in ml required for titration; and

B = weight of the conditioned material taken for test.

Results of duplicate determinations shall be reported.

18. ESTIMATION OF WATER SOLUBLE SULPHATES

18.1 General — This method is applicable to paper containing small quantities of sulphates soluble in water.

18.2 Reagent — The following reagent is required:

a) *Barium Chloride Solution* — approximately 10 percent.

18.3 Procedure

18.3.1 Proceed as described in 17.3.1 for estimation of chlorides.

18.3.2 To the combined filtrate, add one millilitre of concentrated hydrochloric acid, heat to boiling and add dropwise with stirring 5 ml of 10 percent barium chloride solution. Digest at approximately 80°C for 4 hours or longer, filter through a No. 42 Whatman filter paper or equivalent and wash with hot water till free from chlorides.

18.3.3 Transfer the filter paper and contents to a tared crucible, ignite slowly at first with free access of air to prevent reduction of barium sulphate by the filter paper and then at 800°-900°C. Cool and weigh. Carry out a blank test in the same way on the same amount of water and reagents, and subtract any barium sulphate found. Report as sodium sulphate percent.

18.4 Result — Calculate as below:

$$\text{Sulphates (as Na}_2\text{SO}_4 \text{), percent by weight} = \frac{A \times 0.608}{B} \times 100$$

where

A = weight in g of barium sulphate precipitate, and

B = weight in g of material taken for test (see 17.3.1).

Results of duplicate determinations shall be reported.

19. ESTIMATION OF FATTY AND/OR ROSIN ACID EXTRACTABLE BY ETHER

19.1 General — This method covers the procedure for quantitative determination of fatty acid or rosin acid extractable by ether.

19.2 Procedure

19.2.1 Extract a representative portion of the sample, 30 g in weight, with ether in a Soxhlet extraction apparatus for 16 hours. Filter the ether extract, if necessary, and divide into two portions A and B representing 10 and 20 g of the sample respectively.

19.2.2 Total Acidity Content — Distil off the ether from portion A until about 10 ml are left. Add 20 ml of alcohol neutralized to phenolphthalein. Boil the mixture under a reflux condenser for 10 minutes and then titrate with N/20 sodium hydroxide. Carry out a blank experiment side by side and apply necessary correction. Express the result as percent oleic acid present in the sample using the following formula:

$$\begin{aligned} \text{Total acidity (as C}_{17}\text{H}_{33}\text{COOH),} \\ \text{percent by weight} \end{aligned} = \frac{V \times N \times 0.282}{W} \times 100$$

where

V = volume in ml of sodium hydroxide solution,

N = normality of sodium hydroxide solution, and

W = weight in g of the material taken for the test.

19.2.3 Rosin Acid Content

19.2.3.1 Distil off the ether from portion *B* and dry the residue for 30 minutes in a boiling water oven. Add 30 ml of dry petroleum ether (BP-40° to 60°C), boil the mixture under a reflux condenser for 30 minutes and then filter into a separating funnel, the flask and the filter paper being washed with petroleum ether. Wash the solution with 3 successive portions of 20 ml water and then transfer the petroleum ether layer to a flask.

19.2.3.2 Dry the residue remaining after the distillation of the petroleum ether for 30 minutes in a boiling water oven and then treat with 30 ml of methyl alcohol to which has been added previously 2 ml of concentrated sulphuric acid. Boil the mixture under a reflux condenser for exactly 3 minutes. Add 50 ml of 5 percent aqueous sodium chloride solution and transfer the contents of the flask to a separating funnel. Wash the flask 3 times with 20 ml of ether, adding the washings to the contents of the separating funnel. Shake the latter vigorously and return the ether extract layer to the flask.

19.2.3.3 Make two similar extractions of the aqueous portion. Combine the extracts in a separating funnel and wash successively with 20 ml of aqueous 5 percent sodium chloride solution until the washing liquid is neutral to phenolphthalein. Transfer the ether extract to the flask and distil off the ether until 10 ml is left. Add 20 ml of ethyl alcohol previously neutralized to phenolphthalein, boil under a reflux condenser for 10 minutes and titrate with N/20 sodium hydroxide solution.

19.2.3.4 Carry out a similar blank and apply necessary correction.

19.2.3.5 Express the result as percent oleic acid present in the sample using the formula given in 19.2.2.

20. ESTIMATION OF ETHER SOLUBLE MATTER

20.1 General — This method of test covers the procedure for determining quantitatively the ether soluble matter in paper.

20.2 Procedure — Extract 5 g of the material cut into small pieces with ethyl ether in a Soxhlet extraction apparatus of approximately 75-ml capacity, for 4 hours. The ether should siphon from the Soxhlet apparatus approximately 18 times per hour. Filter the extract into a weighed flask and distil off the ether. Dry the residue to constant weight in an oven at 95° to 100°C.

20.3 Result — Calculate the result as percent ether extract on the original weight of the material. Results of two determinations shall be expressed as the matter soluble in ether.

21. ESTIMATION OF BENZENE SOLUBLE MATTER

21.1 General — The method of estimation of benzene soluble matter covers the range of waterproof papers containing bitumen or pitch.

21.2 Test Piece — Cut out a test piece in the form of a rectangle to any convenient size of not less than 100 cm². Measure the sides correct to 0.5 percent for each dimension. Determine the area of the test piece correct to the nearest 0.25 percent of the area. Weigh the test piece correct to 3 significant figures.

21.3 Procedure — Take three test pieces from different parts of a sheet. Shred them together and extract with benzene in a Soxhlet extraction apparatus of 75-ml capacity, for 16 hours. The benzene should siphon from the Soxhlet apparatus approximately 18 times per hour. Distil off the benzene and dry the residue to constant weight at 95° to 100°C.

21.4 Results — Calculate the result as grams per square metre of the original material. At least three determinations shall be made from test pieces cut from widely separate portions of the sample and the highest, lowest and average values reported. The extremes shall not differ from the average by more than ± 5 percent of the average figure.

APPENDIX A

(Clause 13.3.1)

PREPARATION OF MAGNESIUM OXIDE STANDARD

A-1. GENERAL

A-1.1 This method covers the preparation of a magnesium oxide standard for calibrating apparatus used in determining the reflectance and spectral reflectivity of paper and paperboard. The smoke from magnesium freely burning in air, deposited on a satisfactory base, forms a uniform, fine-grained, diffusing surface of a high reflectance. This surface of magnesium oxide (MgO) may be made reproducible; hence, it serves as a convenient and reliable standard.

A-2. MATERIALS

A-2.1 The following materials are required:

- a) *Magnesium* — The magnesium should have high purity and be in either of the following forms: (1) turnings approximately 0.5 mm in thickness and 3 mm in width, preferably of spiral shape, and containing a minimum of magnesium dust; or (2) magnesium ribbon 0.15 mm in thickness and 3 mm in width.

NOTE — Magnesium ribbon is preferred for small surfaces, and magnesium turnings for large surfaces.

- b) *Base* — The magnesium oxide shall be deposited on a surface not affected in air by the heat from the burning magnesium. A satisfactory base may be made of aluminium, block porcelain, sheet steel coated with white vitreous enamel, a baked surface of a sprayed mixture of magnesium oxide powder and distilled water or a block of magnesium carbonate. Milk or opal glass is often unsatisfactory because it easily cracks from heating. Depolished surfaces are better than polished because the oxide adheres better; for the same reason, metallic surfaces are usually to be preferred to non-metallic. Surfaces having reflectance high and uniform throughout the spectrum are better than dark or chromatic surfaces, because with the former a thinner layer of oxide is trustworthy. The thinner layer is desirable, apart from speed of preparation, because it does not chip off so readily. A hollow, tray-like structure is recommended with a flat surface and a rim 1 to 1.5 mm in height to prevent the edges from chipping.

A-3. PROCEDURE

A-3.1 Combustion of Turnings — Place about 5 g of the turnings on a refractory disc approximately 10 cm in diameter and 2.5 cm in thickness with a recess in the top face; vent holes through the disc may be desirable. Zirconium silicate and magnesite are suitable refractory materials for the disc. Ignite the turnings with a hand blow torch or Bunsen burner*. Work the unignited chips beneath the flame with a silica rod until a slowly burning ball or clinker is formed; this gives a steady stream of smoke. When the clinker has to be turned over or broken, in order to permit the magnesium to burn completely, the surface being coated should be temporarily removed, since the burst of flame is likely to carry up large dust particles.

A-3.2 Combustion of Ribbon — Cut the magnesium ribbon in a number of 25-cm lengths. Holding one end of a length of ribbon in pliers, wipe the ribbon with a freshly laundered cloth to remove surface impurities. Ignite the free end with a Bunsen burner* and hold the ribbon in an approximately horizontal position. When the ribbon has burned to a point within about 2.5 cm of the pliers, drop it upon a metal or refractory surface below the work. Burn additional lengths of ribbon until the coating process is completed.

A-3.3 Coating — Place the surface to be coated about 8 to 10 cm above the flame and tilted at 30 degrees to the horizontal. Use of smaller distances

*The operation should be carried out under a well-ventilated hood in order to dispose of the excess oxide. However, the current of air should not be such as to disturb either the flame or the stream of smoke arising from the flame. The air of the room in which the coating is done should be as free as possible of dust and vapours. The operator's eyes should be protected from the high intensities of visible and ultraviolet radiant energy by suitable goggles or other means.

results in a coarse-grained deposit and risks contamination by possible impurities in the magnesium. The flame of the burning magnesium should not be allowed to 'lick' the oxide surface. Move either the combustion disc or the surface being coated from side to side in order to obtain a uniform deposit. Repeat the operation several times until a sufficient deposit is obtained. The layer should be so thick that further increase produces no sensible change in reflectance; the critical thickness is about 0.5 mm. Repeated deposits of thin coatings rather than the full 0.5 mm deposit at one time is recommended. Do not attempt to burn a large number of small charges. In cases where it is inconvenient to measure the thickness of the coating, place a small dot of India ink on the original surface near the edge, then deposit magnesium oxide until the spot cannot be seen in good illumination. If the original surface is dark, put on one coat of magnesium oxide first; a deposit of black smoke (from a candle or smoky gas flame) in a small spot near the edge will then supply a similar test.

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Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022 60 38 43

Southern : C.I.T. Campus, IV Cross Road, MADRAS 600113 235 23 15

†Western : Manakalaya, E9 MIDC, Marol, Andheri (East), BOMBAY 400093 832 92 95

Branch Offices :

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMADABAD 380001 30 13 48

‡Peenya Industrial Area, 1st Stage, Bangalore-Tumkur Road, 839 49 55

BANGALORE 560058

Gangotri Complex, 5th Floor, Bhadbhada Road, T.T. Nagar, BHOPAL 462003 55 40 21

Plot No. 21 Satyanagar, BHUBANESHWAR 751007 40 36 27

Kalaikathir Building, 6/48 Avanashi Road, COIMBATORE 641037 21 01 41

Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001 8-28 88 01

Savitri Complex, 116 G.T. Road, GHAZIABAD 201001 8-71 19 96

53/5 Ward No. 29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003 54 11 37

5-8-56C L.N. Gupta Marg, Nampally Station Road, HYDERABAD 500001 20 10 83

R 14, Yudhister Marg, C Scheme, JAIPUR 302005 38 13 74

117/418 B Sarvodaya Nagar, KANPUR 208005 21 68 76

Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, 23 89 23

LUCKNOW 226001

Patliputra Industrial Estate, PATNA 800013 26 23 05

T.C. No. 14/1421, University P.O., Palayam, THIRUVANANTHAPURAM 695034 6 21 17

Inspection Offices (With Sale Point) :

Pushpanjali, 1st floor, 205-A, West High Court Road, Shankar Nagar Square, 52 51 71

NAGPUR 440010

Institution of Engineers (India) Building 1332 Shivaji Nagar, 32 36 35

PUNE 411005

* Sales Office is at 5 Chowringhee Approach, P.O. Princep Street, 27 10 85

CALCUTTA 700072

†Sales Office is at Novelty Chambers, Grant Road, BOMBAY 400007 309 65 28

‡Sales Office is at 'F' Block, Unity Building, Narasimharaja Square, 222 39 71

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